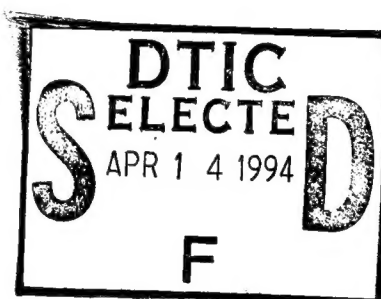


# Assessing Cryogenic Testing of Aggregates for Concrete Pavements

Charles Korhonen and Brian Charest

February 1995



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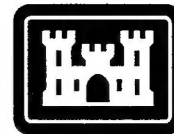
### ***Abstract***

Damage to concrete pavements caused by freeze-thaw deterioration of concrete aggregate remains a serious problem. Current tests for determining an aggregate's freeze-thaw durability can take up to 70 days to perform and results from these tests don't always correlate well with field performance. A rapid test for freeze-thaw durability that would accurately predict field service would be a valuable tool for providing durable concrete. Cycling aggregate 10 times between liquid nitrogen and hot water proved useful as a tool to rule out frost-susceptible aggregate. Pore size distribution measurements reveal pore sizes that are critical to freeze-thaw durability. Aggregates with more than 75% of their measurable pore volume between pore diameters of 0.01 and 5  $\mu\text{m}$  or with more than 95% of their measurable pore volume smaller than 5  $\mu\text{m}$  were susceptible to frost damage. Thus a new freeze-thaw test for aggregates might employ the cryogenic test to screen out all frost-susceptible aggregate and pore size measurement classify the rest. This new test procedure offers results much sooner than current standard test procedures. Further study is needed to refine this method for general use.

For conversion of SI metric units to U.S./British customary units of measurement consult *Standard Practice for Use of the International System of Units (SI)*, ASTM Standard E380-89a, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

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**US Army Corps  
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Cold Regions Research &  
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## PREFACE

This report was prepared by Charles J. Korhonen, Research Civil Engineer, and Brian Charest, Research Civil Engineer, of the Civil and Geotechnical Engineering Research Branch, Experimental Engineering Division, U.S. Army Cold Regions Research and Engineering Laboratory. Funding was provided by the Office of the Chief of Engineers through the Construction Productivity Advancement Research (CPAR) Program, *Rapid Method to Determine Freeze-Thaw Resistance of Aggregate*.

Technical review was provided by Edel Cortez (CRREL) and Steven P. Beck (Michigan Department of Transportation).

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## Assessing Cryogenic Testing of Aggregates for Concrete Pavements

CHARLES KORHONEN AND BRIAN CHAREST

### INTRODUCTION

Despite the remarkable advances made in concrete technology over the past several decades, freezing and thawing deterioration of concrete pavements is still of great concern to state highway officials. The development of air-entrained concrete in 1938 and more recently the introduction of mineral and chemical admixtures to the concrete market have dramatically improved the performance of cement paste. However, there are other important factors affecting the durability of concrete besides those pertaining to the paste. When water in coarse aggregate freezes, it can create sufficient stress within the aggregate itself or within the surrounding cement paste to cause the pavement to crack. Therefore, materials engineers, faced with the problem of finding durable aggregates, have made this a focus of considerable research. Nevertheless, freeze-thaw induced cracking continues to happen on fairly new highways, resulting in extensive and costly rehabilitation.

Usual methods of identifying aggregates able to endure freeze-thaw rely on service records and on laboratory testing. A growing problem with service records is that sources of aggregate known to be freeze-thaw durable are being depleted, particularly in densely populated areas. Added to this, the quality of aggregate from even a proven source can change and environmental laws concerning the mining of aggregates are becoming more restrictive. Thus, laboratory testing is becoming increasingly essential, but the time required to run current test methods frequently exceeds the available design period. A faster and simpler test method would be better.

The Michigan Department of Transportation (MDOT) started freeze-thaw testing of coarse aggregates in 1954 and by 1976 had established a

numerical durability factor that, when used as a criterion for aggregate selection, helped improve pavement performance. The MDOT (1989) freeze-thaw method MTM 115 evaluates the dilation of concrete beams subjected to an environment conforming to ASTM C 666, Procedure B (ASTM 1990). The method cycles nine moist-cured concrete specimens between 4.5 and  $-17.8^{\circ}\text{C}$  and back again by freezing them in air and thawing them in water in 3 hours. Cycling continues until the concrete is exposed to 300 cycles or until it expands beyond a certain limit, whichever occurs first. Much of MDOT's coarse aggregate comes from glacial deposits, which means that physical properties and therefore freeze-thaw characteristics of the aggregate may change from location to location within a given borrow pit. Thus, relying on a freeze-thaw test method that can require up to 70 days to run becomes impractical when answers may be needed each day.

During the fall of 1990, CRREL and MDOT entered into a cost-sharing partnership to explore innovative ways for rapidly differentiating durable from frost-susceptible aggregates. This partnership was made possible through the authority of the U.S. Army Corps of Engineers Construction Productivity Advancement Research (CPAR) Program. Working with assistance from Michigan Technological University (MTU), MDOT was to select aggregates from 20 different sources and conduct conventional freeze-thaw evaluations, concentrating on aggregate moisture conditioning and aggregate size. The CRREL portion was to test samples from the same 20 sources using a proposed accelerated test method that freezes the aggregate particles by immersion in liquid nitrogen and thaws them by immersion in hot water.

**Table 1. Work responsibilities.**

<i>Module</i>	<i>MDOT</i>	<i>MTU</i>	<i>CRREL</i>
I	X	—	—
II	—	X	—
III	X	—	—
IV	X	—	X
V	X	—	—
VI	X	X	X

The overall project consisted of six modules to be completed over 2 years. Table 1 lists the responsible party for each module, for which a brief description is given below.

*Module I*—compare the freeze-thaw performance of concrete to the moisture gain of aggregates tested according to the Iowa Pore Index Test. The Iowa test measures the amount of water injected into aggregate during the first 15 minutes at 35 lb/in.<sup>2</sup> (241 kPa).

*Module II*—test aggregates for differences in absorbed moisture under field and laboratory conditions to develop a standard moisture conditioning procedure for future laboratory testing.

*Module III*—test moisture gain in aggregates conditioned at 100% relative humidity for up to 1 year. This module extends data developed in module II.

*Module IV*—evaluate the freeze-thaw durability of concrete subjected to conventional testing vs. aggregates subjected to cryogenic frost cycling.

*Module V*—evaluate the effect of using 15.2- × 15.2- × 40.6-cm concrete beams and 5.1-cm nominal maximum-size aggregates as opposed to 7.6- × 10.2- × 40.6-cm beams and 2.5-cm nominal maximum-size aggregates in conventional freeze-thaw testing.

*Module VI*—summarize all testing in a report.

## EXPERIMENTAL WORK

This report gives the module IV findings, which compare results of the CRREL rapid freeze-thaw test to the MDOT results from concrete beams tested in the conventional manner.

The original intent of CRREL's module IV was to develop a cryogenic freeze-thaw test procedure that would somehow rank the 20 aggregates from best to worst based on freeze-thaw performance. We later modified this plan during the early stages of testing when we realized that determining how the freezing of interstitial water affects aggregate properties was important for evaluating the cryogenic results. The main expectation was that durability would be controlled by pore structure and

mechanical strength. Consequently, some effort was shifted from solely developing a cryogenic test method to developing pore size distributions, moisture absorptions and crushing strength data. The cryogenic freeze-thaw tests were conducted on the 20 aggregate specimens, but funding limitations allowed us to use only 10 specimens for the remaining tests.

## Test materials

The materials were obtained from 20 sources in Michigan, consisting of gravel, quarried stone and blast furnace slag. The materials came to CRREL in two shipments of 10 samples each. Table 2 provides MDOT data on sample composition and freeze-thaw results from conventional tests.

Sample composition was determined from petrographic analyses of 300 aggregate particles obtained from material retained on each of four sieves from 3/4 in. to no. 4 (4.75 to 19 mm). Freeze-thaw results were obtained from a set of nine concrete beams containing approximately 7% air. All were made with vacuum-saturated aggregate, except for the beams made with the slag aggregate, which was soaked in water for 24 hours. Expansion readings represent the average percentage length change for the set of nine concrete beams per 100 freezing and thawing cycles. The calculated equivalent durability factor is based on beam expansion after 300 freeze-thaw cycles.

MDOT considers concrete beams to be frost damaged when their length has increased 0.10% or when their calculated equivalent durability factor reduces to below 70% after 300 freeze-thaw cycles. Table 2 shows that nine aggregates (3595, 3990, 3035, 4206, 3992, 3791, 3989, 3593, and 3991) produced less durable concrete when evaluated by either the length-change percent or the calculated equivalent durability factor, but that three more (4015, 4141 and 4033) produce less durable concrete by only the calculated equivalent durability factor.

In general, aggregates with poor laboratory performances had higher proportions of sedimentary material while good performers were dominated by igneous material.

## Cryogenic testing

Perhaps the most realistic way to test aggregate is to embed it in mortar and to subject it to the identical wetting and freezing conditions experienced by concrete in the field. In this manner stresses from ice buildup within discrete particles of aggregate and those from water escaping from the aggregate into the surrounding paste model those

**Table 2. Aggregate data by decreasing durability (blast furnace slags listed separately at the end of the table).**

Sample no.	Igneous	Metamorphic	Carbonate	Sedimentary		All others	Expansion per 300 F-T cycles (%)	Calculated equivalent durability factor
				Soft	Chert			
3704	55.2	32.6	0.0	0.0	0.0	12.2	0.000	100
3987	100.0	0.0	0.0	0.0	0.0	0.0	0.000	100
4205	0.0	0.0	100.0	0.0	0.0	0.0	0.015	88
3632	80.9	9.4	2.9	0.0	1.0	5.8	0.024	81
4014	0.0	0.0	100.0	0.0	0.0	0.0	0.030	77
3666	5.8	2.8	91.1	0.0	0.2	0.1	0.033	75
4015	0.0	0.0	100.0	0.0	0.0	0.0	0.048	66
4141	3.7	4.0	91.9	0.2	0.1	0.1	0.072	54
4033	3.0	0.1	96.2	0.2	0.3	0.2	0.081	50
3595	4.1	0.8	94.5	0.0	0.6	0.0	0.105	41
3990	27.8	22.2	45.0	0.8	4.0	0.2	0.138	32
3035	16.2	5.7	72.8	2.4	2.1	0.8	0.150	29
4206	18.9	24.1	50.4	0.6	5.6	0.4	0.159	27
3992	21.7	19.4	51.9	2.0	3.8	1.2	0.174	24
3791	17.0	17.6	56.7	1.5	6.2	1.0	0.189	22
3989	21.6	20.5	49.7	1.5	6.2	0.5	0.309	10
3593	0.0	0.0	99.3	0.1	0.6	0.0	0.402	<10
3991	21.7	19.8	47.1	3.6	6.9	0.9	0.468	<10

	Vesicular	Vesicular dense	Dense	Glassy				
4204	64.1	20.3	14.4	1.2	—	—	-0.003	100
4130	72.4	17.0	9.2	1.4	—	—	0.006	96

of the field situation. Owing to the complexity of accurately simulating field conditions and constraints on time, the MDOT laboratory test method approaches this ideal situation by using small-scale concrete prisms made with a concrete mix similar to that used in the field. It deviates from the ideal by using more rapid freeze rates than those encountered by pavements in nature so as to produce timely results.

The simplest and fastest way to test aggregate is to leave it unconfined, i.e., not embedded in concrete. This avoids the delays and variables inherent with mixing and curing concrete specimens. Though unconfined freezing does not duplicate confinement of aggregate by mortar, the results are usually considered meaningful as relative measures of aggregate quality.

We chose to test aggregates separately by modifying AASHTO T-103 (AASHTO 1978), which describes a rapid procedure for freeze-thaw testing of aggregates. Instead of conventional refrigeration equipment, we chose liquid nitrogen as the freezing medium and instead of room-temperature air, we used hot water for thawing. High freezing rates can be more destructive to specimens of concrete than are low rates. However, in this instance, the freezing rate was not considered a problem as low or high freezing rates on individ-

ual pieces of aggregate act similarly in one important regard: freezing occurs omnidirectionally. Inward freezing inhibits the escape of water by sealing outer pores of the aggregate with ice at the start of freezing. Consequently, volume increases created by freezing of the remaining entrapped water must be elastically accommodated by the aggregate whether freezing is fast or slow.

Aggregate samples were cryogenically tested using the following equipment: a water bath, a dewar for liquid nitrogen, three sieves (1,  $\frac{3}{4}$  and  $\frac{5}{8}$  in. [25, 19 and 16 mm]) and a scale. Aggregate samples were prepared by thoroughly washing, oven drying and separating the aggregate into a size passing the 1-in. and retained on the  $\frac{3}{4}$ -in. sieve. Smaller sized aggregate was also tested and yielded similar results, which are not reported here.

Testing consisted of soaking approximately 500 g of the prepared aggregate in water for 24 hours, followed by freeze-thaw cycling. Each cycle consisted of 1.2 minutes of submersion in  $-196^{\circ}\text{C}$  liquid nitrogen, followed by a 0.5-minute drain period, immersion in 90 to  $100^{\circ}\text{C}$  water for 2 minutes and another 0.5-minute drain period. On the basis of heat transfer calculations, the center of each piece of aggregate cycled from approximately 50 to  $-100^{\circ}\text{C}$  and back again during each 4.2-

**Table 3. Cryogenic frost damage to two groups of 10 aggregate samples in order of decreasing durability.**

<i>Sample</i>	<i>Percent passing</i>	<i>Sample</i>	<i>Percent passing</i>
3666	0.08	3989	0.03
4205	0.10	4015	0.40
3704	1.07	3987	0.60
3791	1.13	3990	0.76
3632	1.43	4014	0.89
4206	1.53	3991	1.69
3593	2.49	3992	1.80
3595	3.94	4033	2.35
4204	33.31	4141	2.74
4130	48.44	3035	2.91

minute cycle. After 10 cycles the aggregate was oven dried at 105°C and resieved.

The resulting freeze-thaw deterioration is reported in Table 3 as the percentage of original weight passing a  $\frac{5}{8}$ -in. sieve. Loss attributable to the mechanical action of sieving was monitored by sieving a control sample not subjected to freezing and thawing.

### Absorption

Aggregates contain a system of absorptive and nonabsorptive pores. The absorptive pores are similar to the capillary pores found in hardened cement paste; they are filled with water by capillary suction. The nonabsorptive pores are like entrained air cavities in concrete that can only be filled by removing the entrapped air and applying an external pressure. The rate at which absorptive space fills with water provides information about frost-susceptibility. Fine pores of high capillarity usually acquire water more quickly than coarse pores of low capillarity. Since freezing water causes distress, aggregate that wets easily and retains moisture strongly is usually more frost-susceptible than aggregate that wets slowly and drains readily. Modules II and III addressed the total absorptive capacity of each aggregate sample by measuring their 1-day through 1-year absorptions. This study examined the initial rate of absorption during the first several minutes of wetting as well as the 24-hour absorption. For reasons discussed earlier, our absorption measurements were made on only half the materials; in this case, those from the first 10 samples.

Our main problem was obtaining a single absorption value for each sample. Two approaches were considered: either constructing a small sample having the identical makeup of the main sample, or selecting individual aggregates from each

sample subgroup and prorating their test results to fit the main sample. We picked the latter approach, as it would provide more potentially useful information.

Absorption readings were obtained by selecting pieces of aggregate, four from each sample subgroup (e.g., four from each of the igneous, metamorphic and sedimentary carbonate groups were tested from sample 3704) and placing them in a wire basket that was submerged in water and suspended from a scale. The weight of water absorbed was recorded every minute. Immediately following each recording, the four pieces of aggregate were momentarily removed from the water to dislodge attached air bubbles.

The resulting weight readings were corrected to account for differences in aggregate size. When water is absorbed into an aggregate, the weight absorbed at any time is proportional to surface area. Thus, for similar materials, absorption rates in larger aggregates having smaller surface-to-volume ratios might be different from smaller ones having larger ratios. To correct for differences in surface area, each reading was divided by the surface area of the four aggregates being tested. (Surface area was estimated, based on spherical geometry, using weight and specific gravity data developed during the mercury intrusion testing.)

The corrected weight readings were then adjusted to make them representative of the entire aggregate sample. Each subgroup result was weighted, according to its portion represented in the overall sample, and added to the weighted results of the other subgroups from the same sample.

Thus, all recorded weight data were corrected to account for surface area variations and adjusted on a weighted basis to be representative of the entire sample. Figure 1 shows how quickly each sample absorbed water when submerged. For example, within the first 6 minutes, samples 3666, 3791, 3595, 4205 and 4206 achieved nearly 10% of their 24-hour saturation, indicating that they might have a relatively fine pore structure (high capillarity). The seemingly coarser grained aggregates showed a more gradual absorption rate. Samples 4130 and 4204, the slags, appear to have the coarsest pore structure. Table 4 ranks the aggregates by fill rate from the fastest to slowest based on the 6<sup>th</sup> minute reading and gives their 24-hour absorptions.

### Crushing value

An aggregate's strength and freeze-thaw durability are both influenced by its internal pore structure. As freezing occurs, damage progresses from internal micro-fissures into larger and larger cracks.

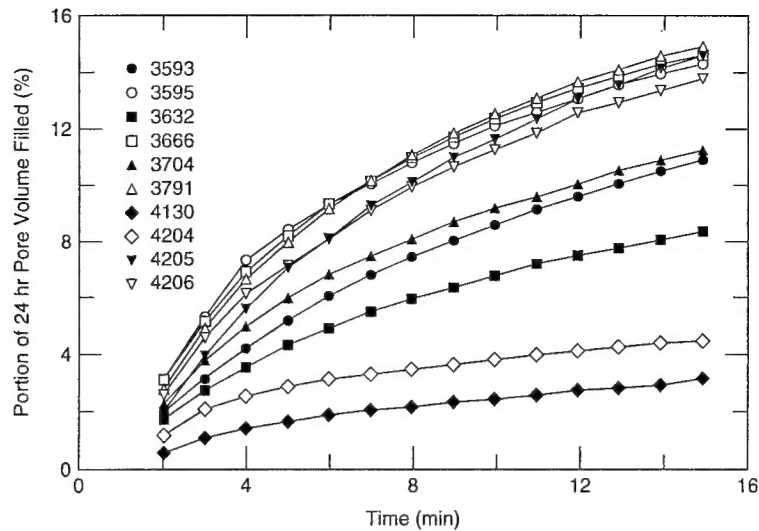


Figure 1. Absorption rates.

Table 4. Ranked absorption readings.

Sample	Percent filled (6 min)	Relative fill rate	Absorption 24-hr (%)
3666	9.3	Fast	1.18
3791	9.1		1.10
3595	9.1		2.00
4205	8.1		0.54
4206	8.1		0.95
3704	6.8	Medium	1.04
3593	6.0		1.75
3632	4.9		1.45
4204	3.1	Slow	2.99
4130	1.8		2.53

Table 5. Crushing value results (%).

Sample	10 F-T*	No F-T
3987	16.1	5.5
3990	17.2	20.6
3992	18.7	16.7
3991	20.2	21.3
2989	22.0	22.3
3035	23.0	24.8
4141	25.9	30.6
4014	27.5	25.5
4033	30.9	37.0
4015	33.8	30.8

\* Freeze-thaw cycles.

In this portion of testing, we compressed aggregates that had been freeze-thaw cycled in a laboratory testing machine in the belief that doing so might complete any damage that freezing had started and thereby manifest the full extent of frost damage.

Our crush test was a modified British Standards Institute (1990) test—BS 812—used to provide information about aggregate strength. The procedure involved placing 500 g of oven dried aggregate, which passed a 1-in. (25-mm) sieve and was retained on a  $3/4$ -in. (19-mm) sieve, into a 3-in. (7.9-cm) diameter steel cylinder. The cylinder was tapped 25 times with a rubber mallet to consolidate the aggregate, and a solid steel plunger was pressed into the top of the aggregate by a 300,000-lbf (1335-kN) Riehle universal testing machine loaded to 3200 lb/in.<sup>2</sup> (22,000 kPa). The aggregate was removed from the test cylinder and sifted through a no. 4 sieve. The crushing value was

calculated by dividing the weight passing the no. 4 sieve by the original weight and was expressed as a percentage.

Material for this portion of testing came from the second aggregate group. Crush testing was done on one set of aggregates after 10 freeze-thaw cycles and on another set with no freeze-thaw cycles. Table 5 shows these results.

Significant variation exists between the control and the freeze-thaw results, suggesting that this procedure is not sensitive enough to aid in distinguishing durable from frost-susceptible aggregate. Perhaps choosing different loading patterns may prove useful in future research.

#### Pore size distribution

Mercury porosimetry was used to characterize the pore size distribution of aggregates from the first sample group. Mercury porosimetry involves injecting high-pressure mercury into the voids of a

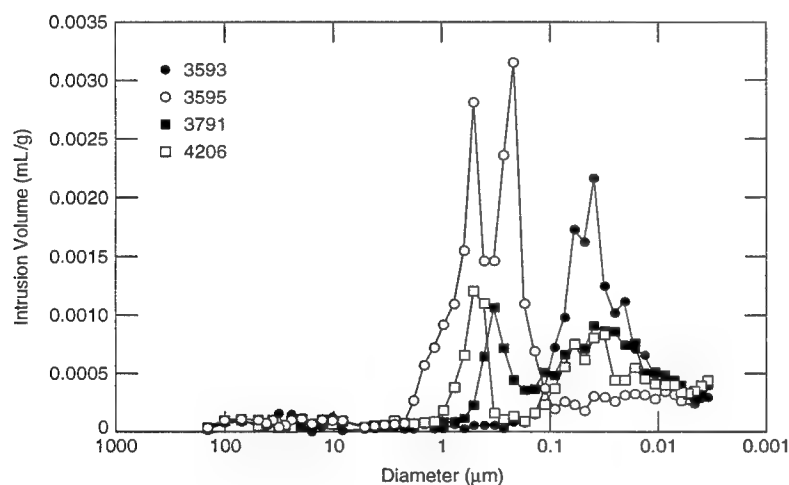
**Table 6. Intrusion volume at given pore diameter ( $\mu\text{m}$ ) and total intrusion volume.**

Sample	<0.005 (%)	0.005 to 0.01 (%)	0.01 to 0.05 (%)	0.05 to 0.10 (%)	0.10 to 0.50 (%)	0.50 to 1.0 (%)	1.0 to 5.0 (%)	5.0 to 10.0 (%)	10.0 to 50.0 (%)	>50.0 (%)	Total (mL/g)
4205	8.6	16.4	28.9	15.0	15.3	3.9	5.9	0.5	3.7	1.8	0.0084
3632	11.4	15.4	27.2	6.3	9.7	3.6	8.1	5.9	11.2	1.2	0.0120
4206	8.2	10.6	29.8	14.1	14.4	17.6	1.9	0.3	2.0	1.1	0.0131
3791	6.8	9.0	35.6	14.2	28.3	2.7	1.0	0.2	1.4	0.8	0.0142
3666	5.2	7.2	16.1	15.3	34.7	5.2	8.1	1.6	4.8	1.9	0.0147
3593	6.3	9.2	47.9	26.6	6.2	0.3	0.2	0.0	2.2	1.1	0.0160
3704	7.3	8.2	14.3	7.1	21.7	5.6	19.4	8.2	6.3	1.9	0.0180
3595	4.0	4.5	8.2	2.5	44.7	24.4	9.8	0.2	1.1	0.7	0.0228
4204	7.1	6.4	7.3	3.5	10.4	5.3	15.1	5.5	28.1	11.3	0.0498
4130	4.6	5.9	12.5	4.3	12.0	4.3	8.8	2.2	18.9	26.3	0.0618

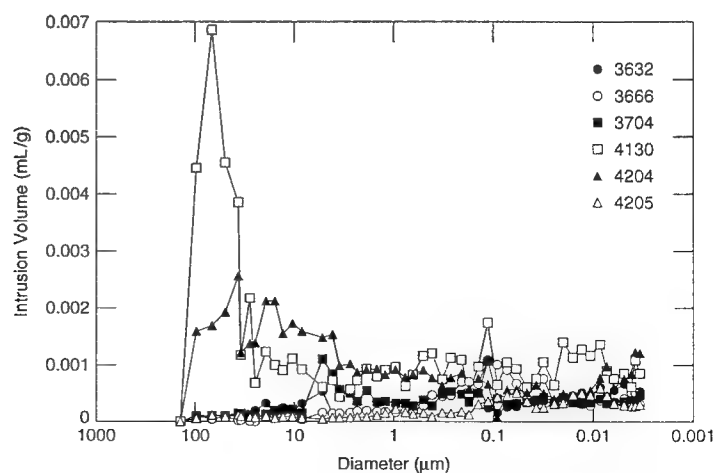
sample to measure its pore volume between an upper and lower limit of pore diameter. Since mercury is non-wetting, pressure is necessary to get mercury into the pores. The pressure required to overcome intrusion resistance is inversely proportional to pore size according to the well-known Washburn equation. In lieu of direct measurements, mercury surface tension and contact wetting angles

were approximated at 480 dyn/cm and  $140^\circ$  respectively. Tests were conducted according to ASTM D4404 (ASTM 1984), except that the aggregates were dried between 100 and  $110^\circ\text{C}$  at atmospheric pressure.

Tests were conducted on four pieces of aggregate from each sample subgroup. Values representative of the entire sample were developed using weight-



*a. Four samples with most of the pore volume below the 5- $\mu\text{m}$  diameter.*



*b. Six samples with more evenly distributed pore volumes.*

**Figure 2. Intrusion volume vs. pore diameter.**

ed averages in the same manner as that employed for the absorption testing.

A pore size distribution analysis yields much data, including total intrusion volume, and the amount of intrusion within any diameter range, as well as sample densities, median and average pore diameters and other information. The entire list of data is not reproduced here, but pore size data abstracted from the results are provided in Table 6, where data are listed in order of increasing intrusion volume. Provided is the total amount of void space as well as the percentage of this space found within certain pore diameter ranges. Figure 2 plots intrusion volume against pore diameter. Clearly, samples 3593, 3595, 3791 and 4206 contain most of their pore volume below 5- $\mu$ m diameter (Fig. 2a), whereas the remaining six samples contain pore sizes throughout the size range (Fig. 2b).

## DISCUSSION

The cryogenic freeze-thaw test was able to distinguish one aggregate from another based on frost damage. By freeze-thaw cycling aggregates 10 times between liquid nitrogen and hot water, it was possible to rank them according to freeze-thaw damage within an hour, as opposed to the months that it now takes with current freeze-thaw test methods.

Table 7 and Figure 3 compare the cryogenic freeze-thaw results to those of MDOT. The first impression that one might gather from comparing the individual rankings in the table is that the two freeze-thaw methods do not correlate well. This is most evident by looking at the results for the two

**Table 7. Comparison of aggregate durability\* obtained using conventional freeze-thaw techniques to that using the cryogenic method.**

MDOT rank	CRREL rank	Sample	MDOT calc. equiv. durability factor	CRREL percent passing
1	5	3987	100	0.60
2	8	3704	100	1.07
3	19	4204	100	33.31
4	20	4130	96	48.44
5	3	4205	88	0.10
6	10	3632	81	1.43
7	7	4014	77	0.89
8	2	3666	75	0.08
9	4	4015	66	0.40
10	16	4141	54	2.74
11	14	4033	50	2.35
12	18	3595	41	3.94
13	6	3990	32	0.76
14	17	3035	29	2.91
15	11	4206	27	1.53
16	13	3992	24	1.80
17	9	3791	22	1.13
18	1	3989	10	0.03
19	15	3593	<10	2.49
20	12	3991	<10	1.69

\* Expansion readings were omitted since their ranking is identical to that from the calculated equivalent durability factor.

slags, 4204 and 4130. MDOT ranked these aggregates 3 and 4 while CRREL ranked them 19 and 20.

The blast furnace slags performed very poorly in unconfined testing but quite well in the standard laboratory freeze-thaw test. The reason for this may be explained by their wetting characteristics. Most researchers would agree that aggre-

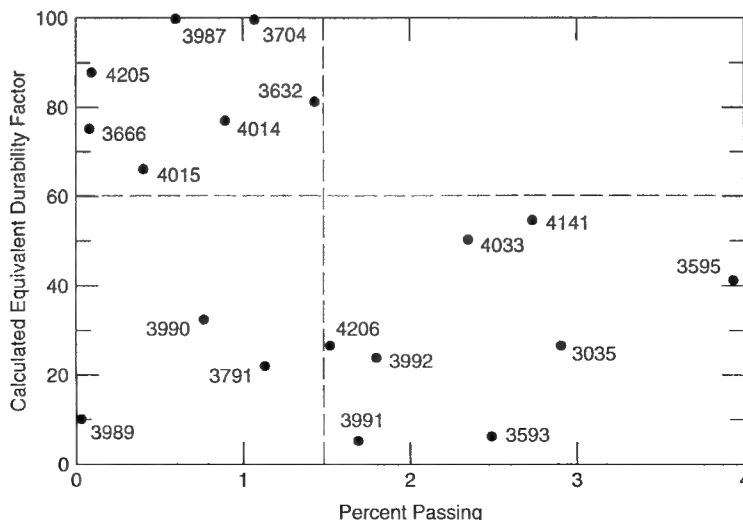


Figure 3. Calculated equivalent durability factor vs. percentage passing.

gates, such as the two slags, having high 24-hr absorptions (Table 4) would have a high potential for reducing the freeze-thaw durability of concrete. The slags' surfaces, honeycombed with visible pores and cavities, can readily fill with water when in direct contact with bulk water, reducing performance in the cryogenic test. However, in concrete the large pores in the slag may act like bubbles of entrained air that can remain empty for long periods, even when surrounded by water-saturated paste. Indeed, aggregates with large pores can contribute to the overall frost resistance of concrete by providing functional relief space into which water can escape during the formation of ice in the surrounding cement paste. As previously discussed, the slag was not vacuum-saturated.

This would suggest that the slag was not saturated and may perform very well in concrete until it becomes critically saturated, which may take a considerable amount of time, but that after becoming critically saturated deterioration would be swift. Further study is needed to confirm this hypothesis.

Once the two slags are dismissed, the two test methods correlate closer, though they are not in full agreement to one another in distinguishing durable from frost-susceptible aggregate. Figure 3 shows this. While there is significant data scatter, two facts emerge—no aggregate with a calculated equivalent durability factor above 60 in the conventional test had more than 1.5% passing in the cryogenic test and all aggregate with more than 1.5% passing had a calculated equivalent durability factor below 60%. The two methods disagree

for the aggregate in the lower left quadrant of Figure 3, which have low calculated equivalent durability factors and low percent passings. Thus, the cryogenic test was best at identifying frost-susceptible aggregate. However, as discussed next, pore size measurements offer the possibility that the questionable aggregates, those in the lower left quadrant of Figure 3, can be properly identified without having to resort to the tedious conventional testing.

Typically, aggregates that have large pore volumes and high amounts of fine pores are likely to be susceptible to frost damage. According to Table 6, sample 3791, the only one of the three samples tested for pore size and located in the "questionable" lower left quadrant in Figure 3, contained over 95% of its measurable pore volume in pores of diameters smaller than 5  $\mu\text{m}$  (5  $\mu\text{m}$  was shown to be a natural pore size delimitator in Fig. 2). Conversely, aggregates with high amounts of coarse pores are usually durable. The aggregates in the "durable" upper left quadrant of Figure 3 that were tested for pore size (4205, 3666, 3632, 3704) contained significantly more intrusion volume in pores of diameters greater than 5  $\mu\text{m}$  than was in 3791, as shown in Figure 4.

Figure 4, a plot of pore volume fractions vs. calculated equivalent durability factors, demonstrates the close relationship between these two parameters when 5  $\mu\text{m}$  is used as the benchmark. Expressly, as pore structure becomes finer (>95%) durability decreases and as pore structure coarsens (<95%) durability increases. The reader will recall that the two slag samples, 4130 and 4204, were not amenable to the cryogenic test but were

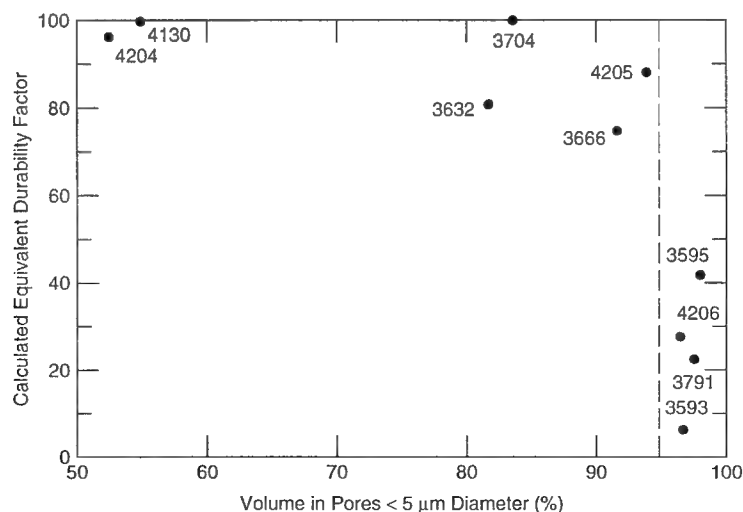


Figure 4. Calculated equivalent durability factor vs. volume percentage of measurable pores less than the 5- $\mu\text{m}$  diameter.

durable in the conventional test and they show up as having the coarsest pore structure, i.e., the highest intrusion volume, in Figure 4.

From the foregoing discussion, we propose that an improved freeze-thaw test might employ the cryogenic test to screen out frost-susceptible aggregates and use the pore size measurement technique to identify aggregates containing favorable pore-size distributions from those aggregates passing the cryogenic test. However, in light of the limited testing in this study, we consider the proposed test unsuitable for immediate application to all aggregate types without further work to determine exactly how the internal pore structures of aggregates affect freeze-thaw performance.

To take the importance of pore size one step further, the literature suggests that certain pore size ranges are more critical than others to freeze-thaw resistance. This is partly because, as pores become smaller, the temperature at which pore water freez-

es in them decreases to the point where pores become too small for water to freeze at all. Large pores tend not to fill with water, so freezing is not a problem in them. Thus, there should be an upper and lower size range beyond which durability is not affected. Mindess and Young (1981) indicate that water will not freeze in 0.01- $\mu\text{m}$  pores until the temperature drops to  $-5^{\circ}\text{C}$  and in 0.0035- $\mu\text{m}$  pores until  $-20^{\circ}\text{C}$ . Further, Neville (1981) shows that, in cement gel possessing pores smaller than 0.001- $\mu\text{m}$ , ice cannot form until  $-78^{\circ}\text{C}$ . On the basis of this information and under the assumption that the migration of water between pores is not a factor, which it may be, it seems that pores smaller than 0.0035  $\mu\text{m}$  should not affect durability as water in them will not freeze at ordinary winter temperatures.

Intrusion values for each aggregate sample were separated into several groupings to see if there was a "critical" pore range affecting durability. In sort-

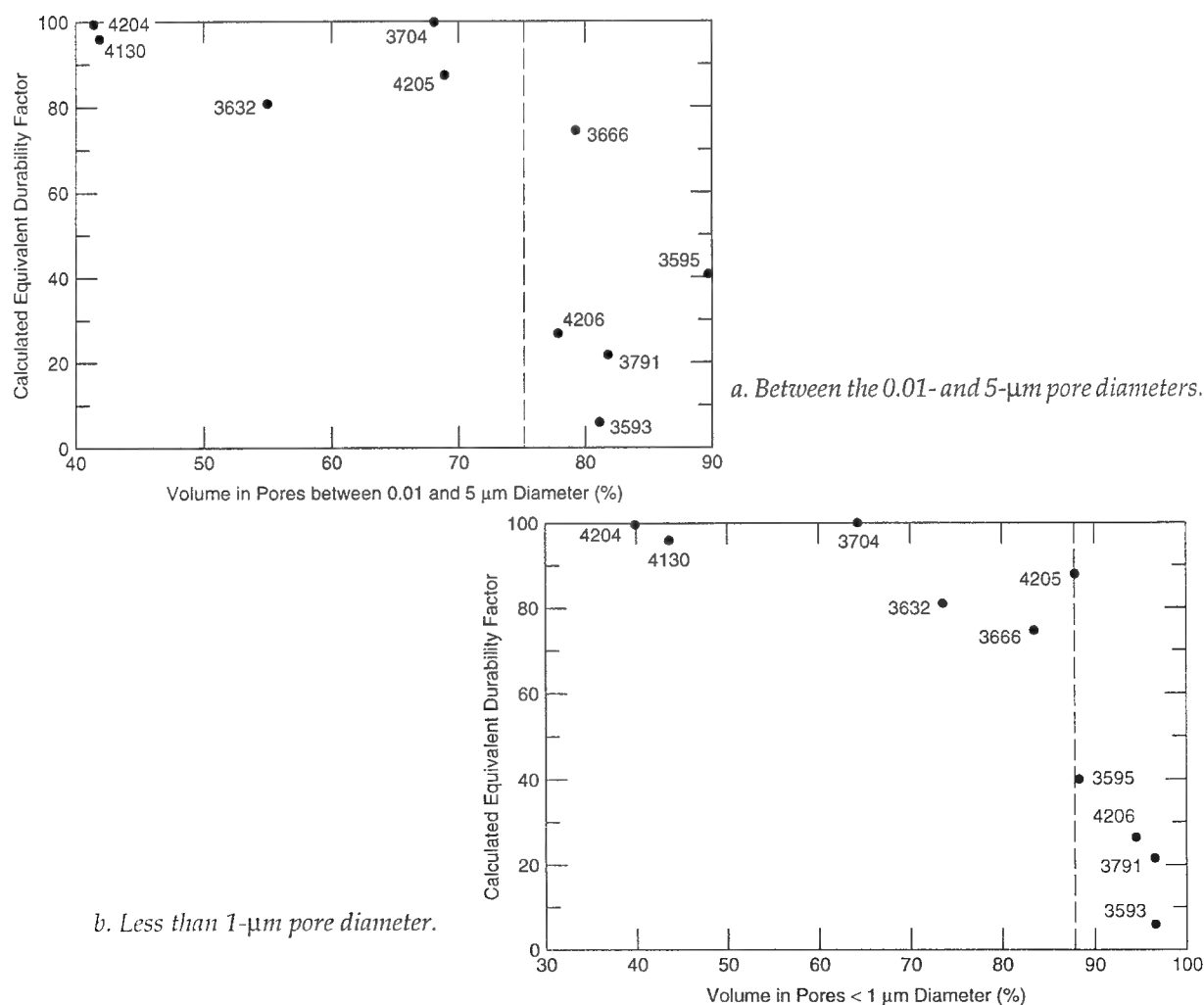
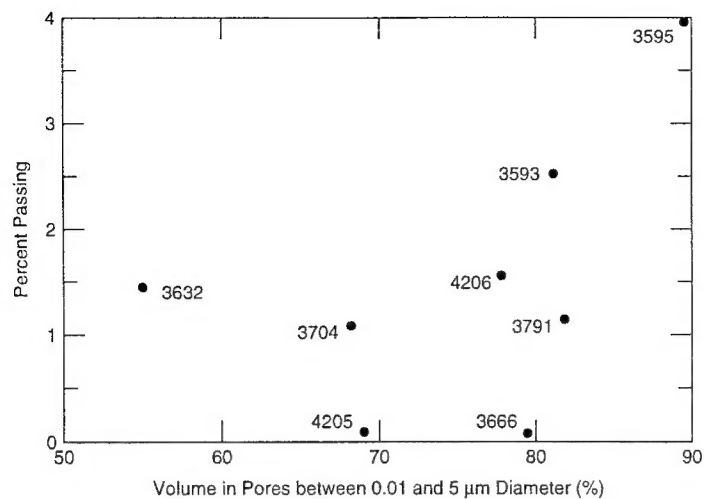
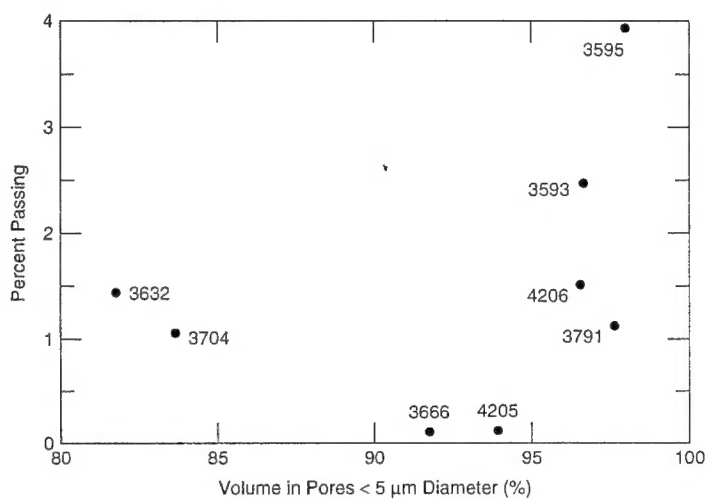


Figure 5. MDOT durability factor vs. percentage of pore volume.



*a. Between the 0.01- and 5- $\mu\text{m}$  pore diameters.*



*b. Less than 5- $\mu\text{m}$  pore diameter.*

Figure 6. CRREL percentage passing vs. percentage of measurable pore volume.

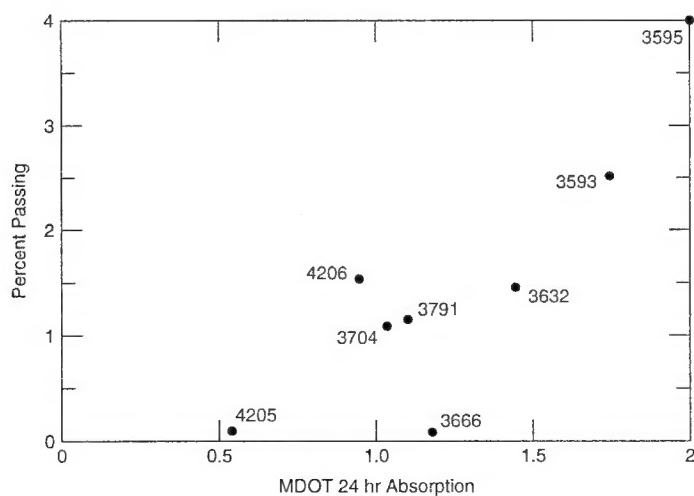


Figure 7. CRREL percentage passing vs. 24-hour absorption.

ing through the many possible ranges, 0.01 to 5  $\mu\text{m}$  (Fig. 5a) seemed to produce a good correlation with the durability factor, suggesting that this mid-range of pore sizes is important to conventional freeze-thaw tests. That is, pores smaller than 0.01 and larger than 5  $\mu\text{m}$  are little affected by the low temperatures imposed upon them. Except for 3666, aggregates with more than 75% of their measurable pore volume in this range are frost-susceptible.

A pore range that relates pore diameters to durability factors about as well as the one shown in Figure 5a is that for pore sizes less than 1  $\mu\text{m}$ . Figure 5b shows that, except for 4205 being borderline, aggregates with more than 88% of their pore volume in this range are frost-susceptible. (Table 6 shows that aggregate 4205 has a very low total intrusion volume, which would tend to limit the effect.)

The same pore ranges were compared to the cryogenic test results. Figure 6 plots percentage passing against percentage of total intrusion between 0.01 and 5  $\mu\text{m}$ , and less than 5  $\mu\text{m}$ . Though percentage passing tends to increase with percentage of total intrusion, the correlation to durability factor is not as good as it was with the conventional test results. Even less correlation existed in the less than 1- $\mu\text{m}$  range, so this plot is not shown.

Additional deterioration because of ice forming in small pores at the extremely low temperature used in the cryogenic test may explain the poor correlation between pore size and measured frost damage. However, the pore size analysis could not accurately measure pore volumes below the smallest pore freezable (0.0035  $\mu\text{m}$ ) in the conventional test to confirm this supposition. Possible ice accretion is another explanation. The cryogenic test causes pore water to freeze in minutes, whereas in the conventional test, freezing takes place over a period of an hour or more. The slower the freezing rate, the more chance water has to migrate from smaller pores into larger pores, and the greater the possible deterioration. Without detailed measurements, this also is difficult to substantiate.

Absorption values have been suggested in the past as being a possible indicator of relative freeze-thaw resistance of aggregates. Normally, aggregates with the lowest absorptions are believed to make the most durable concrete. Figure 7, which compares 24-hour absorption values to CRREL percent passing values, shows an unmistakable trend of decreasing durability with increasing absorption. A similar trend existed when absorptions were plotted against durability factors. However, data scatter in the mid-range of absorption values

is too great to provide usable information in either the cryogenic or the conventional tests. More testing of statistically larger sample sizes is needed to develop this possible relationship between early age absorption and durability.

## CONCLUSION

We assessed cryogenic frost cycling as a method for testing the freeze-thaw resistance of aggregates as part of a cooperative project with MDOT. The cryogenic freeze-thaw test was able to differentiate among a series of 20 aggregates on the basis of frost damage. There was not a one-to-one correlation in durability rankings between this test and the conventional test. However, all aggregates identified as frost-susceptible by the cryogenic test were deemed frost-susceptible by the conventional test.

Pore size distribution measurements reveal a pore range critical to freeze-thaw durability. Aggregates containing more than 75% of their total pore volume between 0.01 and 5  $\mu\text{m}$  were nearly always frost-susceptible. A somewhat better result was found for aggregates containing less than 5% of their total pore volume in pores of greater than 5  $\mu\text{m}$  in diameter.

On the basis of the limited testing done in this study, the cryogenic freeze-thaw test appears to have potential as a very rapid test for identifying aggregate that should not be considered for highway use. For those aggregates that pass the cryogenic test, it appears that some sort of pore-distribution measurement, such as high-pressure mercury porosimetry, should be used to single out freeze-thaw durable aggregate acceptable for highway pavement. The standard ASTM C666 test may only be resorted to if time allows.

These conclusions are considered valid for the aggregates tested. More research is needed to determine the suitability of this test combination for general use.

## NEEDED RESEARCH

The performance of aggregates exposed to freezing and thawing depends heavily on pore size distribution and porosity. Though others have studied these issues, a better understanding of this interrelationship is needed to give us confidence in developing improved freeze-thaw test methods. The following are proposed research areas.

1. Examine rapid freeze-thaw testing of unconfined aggregates using other temperature ranges.

The extremely low temperature used in this study may have been much more destructive than freezing at a higher temperature would be.

2. Examine various pore volume measurement techniques, including mercury intrusion porosimetry. Absorption rates and total absorption volumes measured under differing conditions have the potential for providing useful information on pore sizes related to freeze-thaw issues.

3. Examine pore blocking materials to eliminate the negative effect of certain pore ranges. The pores shown to have a detrimental effect on durability have high capillary attraction and thus may be easy to impregnate with fluids or gels unaffected by freezing. This could be particularly useful in repair situations where only small amounts of aggregate need treatment.

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# REPORT DOCUMENTATION PAGE

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